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6. AUTHOR(S)
Henry W. White

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UNIVERSITY OF MISSOURI
COLUMBIA, MO 65211

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13. ABSTRACT *(Maximum 200 words)*

Two facilities were constructed for synthesis of protective thin films. The first system utilized the recently developed Laser Absorption Wave Deposition (LAWD) technique. With this technique multiple layers of ultrahard carbon films can be grown directly on a variety of surfaces. A pulsed infrared laser is used to create a highly ionized plasma in an atmosphere of a gas mixture, such as methane in hydrogen, into which a plume of ablation particles from a suitably placed target are brought. The ablation particles can be coated and deposited on a substrate. The second system utilized Electron Cyclotron Resonance Plasma Enhanced Chemical Vapor Deposition (ECR-PECVD). This technique can be used to grow a wide variety of thin films using a microwave plasma source. The energy of arriving adatoms varies from a few to several hundred eV. Films will be grown and characterized under a three year, separately funded ARO program for the growth of protective films on structural substrates such as Fe, W and Ta.

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GROWTH FACILITY FOR NANOCOMPOSITE FILMS

FINAL TECHNICAL REPORT

BY

HENRY W. WHITE

PRINCIPAL INVESTIGATOR

SEPTEMBER 29, 1996

U.S. ARMY RESEARCH OFFICE

GRANT NO. DAAH04-93-G-0479

UNIVERSITY OF MISSOURI

COLUMBIA, MO 65211

STATEMENT OF THE PROBLEM

Funds were requested to construct facilities for growth of nanostructure films using two techniques: (i) Laser Absorption Wave Deposition (LAWD), and Electron Cyclotron Resonance Plasma Enhanced Chemical Vapor Deposition (ECR-PECVD). These two facilities would be used to grow protective films on structural materials for characterization.

With the recently developed LAWD technique, multiple layers of ultrahard carbon films can be grown directly on a variety of surfaces. A pulsed infrared laser is used to create a highly ionized plasma in an atmosphere of a gas mixture, such as methane in hydrogen, into which a plume of ablation particles from a suitably placed target are brought. The ablation particles can be coated and deposited on a substrate.

The ECR-PECVD technique can be used to grow a wide variety of thin films using a microwave plasma source. The energy of arriving adatoms varies from a few to several hundred eV. Films grown in the combined LAWD and PECVD facility will be characterized under a three year program that was presented in a companion proposal to the ARO, Material Sciences Division. That proposal was funded, the focus of which is the design and growth of protective films on structural substrates such as Fe, W and Ta.

SUMMARY OF THE MOST IMPORTANT RESULTS

Construction of a Laser Ablation Wave Deposition (LAWD) Film Growth System:

In LAWD film growth, an intense infrared laser pulse is focussed above a surface and avalanche breakdown occurs in the gases above the surface.[1,2] A schematic of the LAWD process is shown in Fig. 1. A combined ionization-shock wave front then propagates in both the up-beam and down-beam directions. Within these wavefronts, ion densities may approach 10^{20} cm^{-3}, pressures may reach 20 kbar, temperatures 10^5K and mass fluxes near 10 kg/cm^2-sec.[3,4] The surface is also transiently heated by the incident beam. LAWD should be viewed as a gas phase process, in which the gas phase and the substrate phase become mixed. It utilizes energy transport mechanisms, rather than diffusional or mass transport processes associated with conventional CVD or ablation processes. As such, LAWD can be considered a transient high pressure, high

temperature technique--good for growth of films such as ultrahard carbon and other films.

A Continuum Nd:YAG laser with up to 200 mJ per pulse and a repetition rate variable from a few Hz up to 50 Hz was selected as the pulsed source. An optical rail for focusing the beam to obtain an irradiance of approximately 10^{11} W/cm^2 was constructed. A small chamber with a rotating target holder was constructed in the Physics Machine Shop. Gas flow metering and pressure monitoring equipment was added. All instruments were computer controlled using LABVIEW. A schematic of the LAWD system is shown in Fig. 2.

Construction of a ECR Plasma Enhanced CVD System:

PECVD can be used to grow a variety of thin films. A number of excellent reviews are available.[5] While many studies have been done, much work remains before an acceptable level of understanding is reached on the relationships between adatom flux at the substrate, adatom energy, substrate temperature, types and concentrations of ions and molecular species in the gas phase, plasma electron density, etc. and how these parameters relate to the properties of the films grown.

An ECR microwave source manufactured by WAVEMAT, Inc. was chosen. This source has magnetic field lines parallel to the substrate sample (circular to the axis of gas flow). It was hoped that this geometry might give superior film growth. The supporting ideas were that high energy electrons would not be driven along magnetic field lines into the growing film surface, creating damage. Overall, this feature would allow the substrate to be placed close to the plasma ball, with the possibility for exposure to a dense, low-energy plasma without surface damage. A custom chamber with optical ports, a large turbopump (450 l/s) with dual stages for high pressure pumping was chosen to allow high flux of desired adatoms at the substrate, to avoid defects due to adatom starvation. Flowmeters, pressure monitoring and regulating instrumentation were also added. A schematic of the ECR-PECVD system is shown in Fig. 3.

New Research Areas Relevant to DoD Interest

Several new research areas relevant to DoD interest have been generated by construction of the two facilities for thin film synthesis. These include hard coatings, corrosion resistant barriers, and thermal shock and oxidation resistance films for ultrahigh temperature applications.

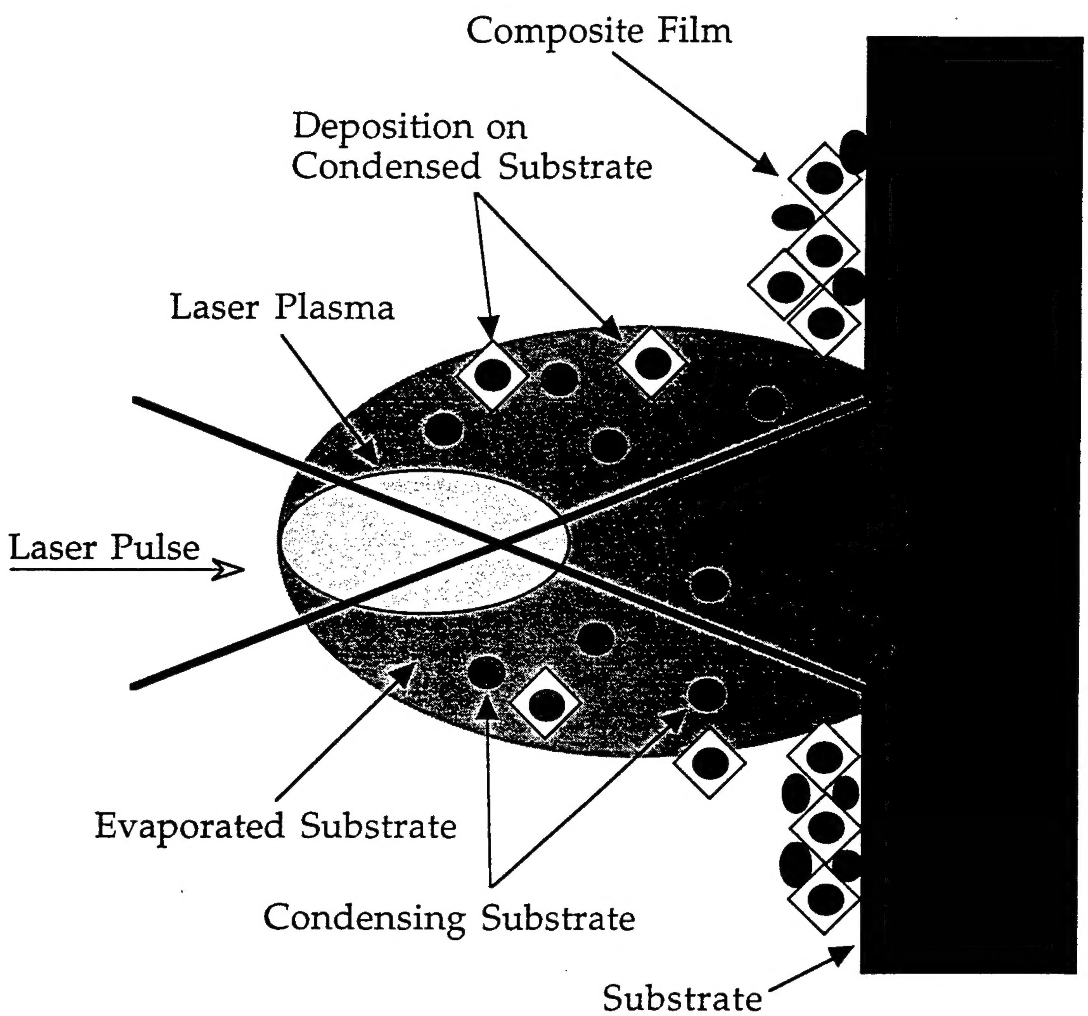

Fig. 1. Schematic of the LAWD process.

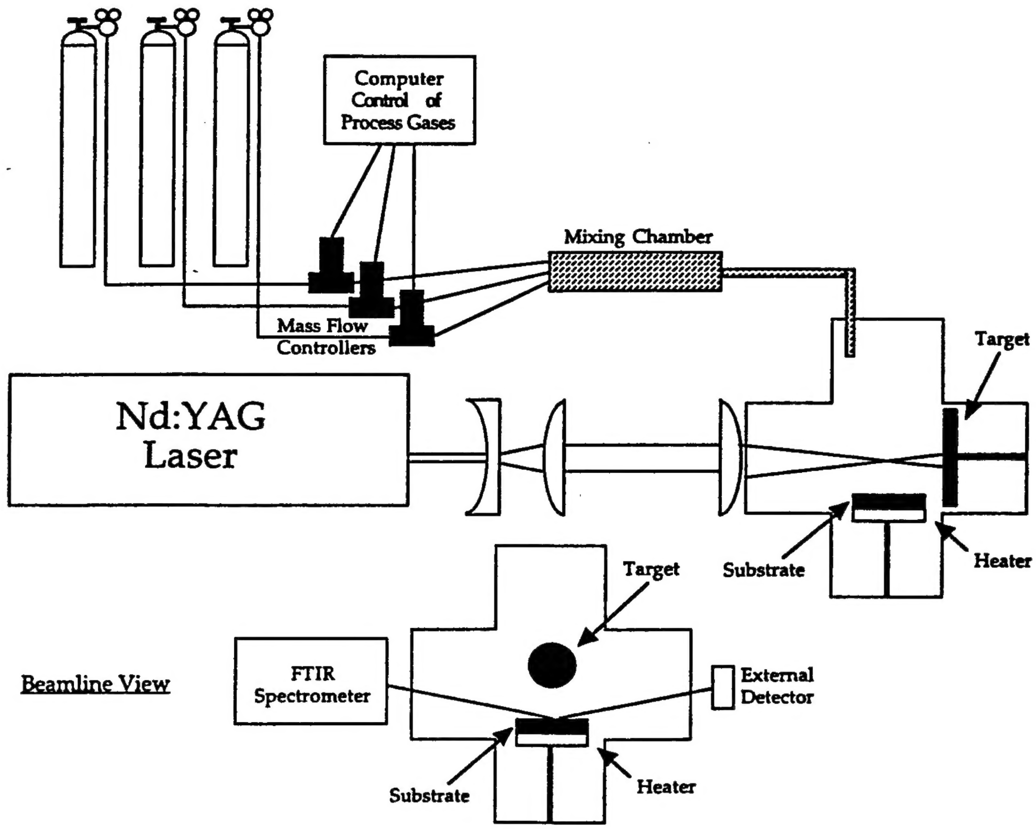

Fig. 2. Schematic of the LAWD facility for film growth.

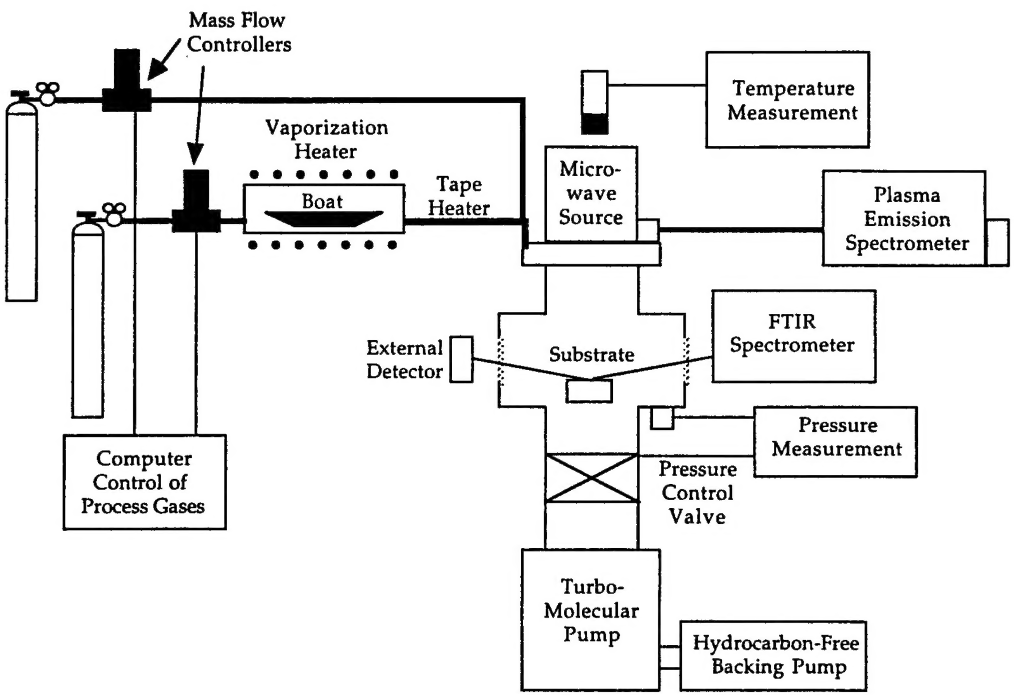

Fig. 3. Schematic of the ECR-PECVD facility for film growth.

LIST OF ALL PUBLICATIONS AND TECHNICAL REPORTS

None

REPORT of INVENTIONS

None

LIST OF ALL PARTICIPATING SCIENTIFIC PERSONNEL WITH ANY ADVANCED DEGREES EARNED BY THEM WHILE EMPLOYED ON THE PROJECT

Henry W. White,. Principal Investigator

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